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A METHOD TO OBTAIN AN ULTRACLEAN ENVIRONMENT

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ABSTRACT: The Goddard Space Flight Center has designed and fabricated a system which offers an effective approach toward solving the problems usually encountered in creating an ultra high vacuum. The vacuum techniques described find application in an Auger Spectrometry System.

This ionization pumped system is unconventional in that; (1) Interior chamber cryogenic shrouding is eliminated. In lieu of the internal shrouds the method is to submerge the entire chamber exterior in a dewar of liquid nitrogen. Eliminating the shrouds within the chamber eliminates sources of contamination and the exposing of the wall exterior to the cryogenics eliminates the typical ambient temperatures that chamber walls normally experience, (2) The ionization pump is located within the ultrahigh vacuum chamber, rather than outside. This eliminates the gate valve typically associated with an externally mounted pump and permits greatly increased realization of the conductance pump capacity. The pump is protected by use of gas purging when pressures are increased to atmospheric.

The method of precleaning, prior to bakeout, involving high temperature gas purging is described as well as the entire systems operation. Pressures in the order of 7×10^{-13} torr have been observed.

KEY WORDS: vacuum, pumping, ultraclean, cold shrouding, environment, contamination, low temperature, low pressure, ion pumping, anti-migration traps.

A METHOD TO OBTAIN AN ULTRACLEAN ENVIRONMENT

An ultraclean environment is often mandatory in space oriented research, particularly when experimental materials must be free of

surface contamination. The Goddard Space Flight Center has designed two ultra high vacuum systems using an effective approach to solve some of the problems usually encountered while attempting to create an oil-free environment. One of these systems is used to develop ultra high vacuum components and techniques. The other system is used for Auger Spectrometry research. The spectrometer can detect partial monolayers of materials and identify these materials. This unique system is one example clearly demonstrating the need for a noncontaminating experimental environment. Since an oil free environment for this Auger Spectrometer was mandatory, an ionization pumped system[1] was developed, which will be described here.

Conventionally, ion pumped ultrahigh vacuum systems employ an internal optically dense liquid nitrogen shroud[2] in conjunction with a titanium pump, Figure 1(A). The ionization pump is mounted external to the chamber and isolated by a gate valve. Typically, the gate valve utilizes an elastomer[3] O-ring. While this geometry and selection of components finds wide acceptability, improvement can be realized by locating the ionization pump inside the chamber, Figure 1(B); by eliminating the gate valve and the cyro shrouds and by slightly changing the typical pump down technique.

ULTRAHIGH VACUUM CHAMBER

The entire vacuum system, Figure 2, is constructed from metal and all seals are metallic. Ceramic to metal feedthroughs are used. Within the cylindrical chamber (20-inch diameter by 36-inch high) are the ionization pump, titanium sublimation pump, an electron trap and shelves for mounting experiments. Thermocouples monitor the chambers' wall at four separate elevations. The work area of the chamber is top loaded through a choice of metal sealed orifices up to 18 inches in diameter. The dome includes an observation port and a high intensity light port.

Surrounding the vacuum chamber on the sides and bottom is a metal thermos bottle. The annulus between the chamber and the thermos houses heater elements and when the heaters are activated, this area becomes an oven for bakeout. The area is also capable of being filled with liquid nitrogen, thus insuring the walls of the chamber being uniformly cooled to LN_2 temperature. Cryogenics are not allowed to contact feedthroughs directly, they are cooled through conduction. LN_2 can be gravity drained through a valve in the bottom of the thermos.

[1, 2, 3]Numbers in parentheses refer to the list of references appended to this paper.

Optically baffled titanium pump filaments are mounted through the center bottom of the chamber. When used, titanium is vaporized onto the LN_2 cooled walls of the chamber providing an intrinsic pumping speed in the order of 60,000 1/sec (approximately 60 $1/in^2/sec$).

A 250 1/sec ionization pump is located above the titanium pump well, a position near the center of the chamber vertically. Conductance for the ion pump far exceeds its capacity and the solid angle of view it possesses is much improved over external mounting techniques.

An oven hood covers the top of the chamber and all feedthroughs during bakeout cycles. It is planned to seal this hood to the system at a later date so the oven/liquid nitrogen area can be used for a third beneficial function, a differently pumped area completely surrounding the vacuum chamber and feedthroughs. Currently, this feature does not exist.

GAGES

Three separate gages were used to evaluate the chambers' performance during the equipment checkout phase and installation. The location of two of these is shown in Figure 3. These included the ionization pump gage which reads pressures to 1×10^{-10} torr, a cold cathode-type gage and a hot cathode gage which cathode gage which read to 1×10^{-13} torr. The hot and cold cathode gages are mounted at the top of the chamber. As one might expect, variations in pressure readings were observed, however these were well within an acceptable range. Pressures presented in this report are from the hot cathode gage.

PUMPS

Five different types of pumping mechanisms are used in this system: a mechanical pump, a sorption pump, an ionization pump, a titanium pump and liquid nitrogen cooled surfaces which pump considerable gases and vapors.

The mechanical pump and the sorption pump are used for roughing the chamber. The mechanical pump also evacuates the double walled thermos bottle. Dry nitrogen is admitted into the chamber to maintain a pressure of 200 microns while roughing with the 15 cfm mechanical pump. This assures only viscous flow during the first stages of roughing. Roughing is completed by valving out the mechanical pump and the nitrogen gas supply and then activating the sorption pump.

The ionization pump, titanium pump and liquid nitrogen cooled walls are used to evacuate the vacuum chamber to lower pressures.

By location the ionization pump inside the vacuum chamber, the typical gate valve can be eliminated.

When rapid re-cycling of the chamber from atmosphere to ultra high vacuum is required, a feature not normally expected of an ultra-high vacuum system, elimination of the gate valve can offer some advantages. All gate valves decrease the conductance and reduce the throughput of a dynamic system. They also present additional surfaces to be cleaned and where elastomer O-rings are used, bakeout temperatures are often restrictive. Protection of the ion pump from contamination is accomplished by special operating procedures which are strictly adhered to. Venting the system to dry nitrogen while the system temperature is in the order of 125°C is necessary. The system is never allowed to see atmospheric pressure for over 15 minutes. Shorter exposures are more the rule.

BAKEOUT

All surfaces exposed to the ultra high vacuum are baked with good uniformity to 450°C. When it is desired to obtain a very low pressure (less than 1×10^{-13} torr), the chamber is cooled over a period of around eight hours so that all surfaces are cooled at nearly the same rate. Heat is added in ever decreasing increments during the cool down cycle to achieve this goal. When the chamber temperature approaches 50°C, liquid nitrogen is introduced until the entire chamber is submerged. By reducing the wall temperature the vapor pressure of all materials located thereon is reduced, consequently, even those gases exhibiting virtually no partial pressures on a mass spectrometer are affected. The mild bakeout, 450°C is very effective when the chamber is cooled to LN_2 temperature.

Since typical cryogenic shrouds are eliminated, their problems go with them. They are difficult, if not impossible, to bake out or cool uniformly. Reduction of surfaces exposed to the ultrahigh vacuum is of prime importance, to eliminate sources of contamination.

FORELINE TRAPS

Two traps are used in the foreline. A bakeable activated absorbent trap is located next to the mechanical pump. This trap eliminates much of the oil backstreaming from the mechanical pump. It eliminates some of the oil migration. To completely trap oil from the mechanical pump, the second trap employs a removable and throw-away section, Figure 4, so that oil cannot migrate through it when it is at ambient temperature. The second trap is activated with liquid

nitrogen such that oil cannot migrate through it when it is charged. In all cases, this trap is exposed to viscous flow in the direction of the mechanical pump, a feature which inhibits oil backstreaming.

Mechanical pump preliminary evacuation to 200 microns is an important feature of the operating procedure. Mechanical pumps evacuate all gases and are not selective as are sorption pumps.

OPERATIONAL SEQUENCE

Initial roughing of the chamber is accomplished with the double trapped mechanical pump to a controlled pressure of around 200 microns. Dry nitrogen is metered into the chamber to prevent the mechanical pump from evacuating the chamber to a lower pressure. Additional evacuation is completed with the sorption pump while the mechanical pump and gas bleed are valved out of the system. The entire evacuation procedure and sequence is performed after the chamber has been brought to at least 150°C. Roughing with the mechanical pump should continue for at least ten minutes. When the chamber pressure is below 10 microns, the sorption pump is valved out and the ionization pump is started. (The pressure can be reduced to around 1×10^{-4} torr with liquid nitrogen prior to starting the ionization pump if the pump seems hard to start.)

Within a few minutes, the ionization pump will have pumped the chamber to a pressure of 5×10^{-7} torr or lower. To achieve lower pressures, a bake cycle is initiated. Figure 5 illustrates a pump down cycle achieve with the Auger Spectrometry system. The newer system currently being evaluated is expected to better the pump down cycle exhibited by the first system.

ENVIRONMENT ACHIEVED

The initial cleaning of the system involved a long time (72 hours) bakeout. Normally a four hour bakeout will be sufficient to enable the chamber to achieve a vacuum in the 10^{-11} torr range or lower. The predominating gas species at 10^{-11} torr are H_2, CO and He. In the 10^{-13} torr range H_2 and He were the dominant constituents.

REFERENCES

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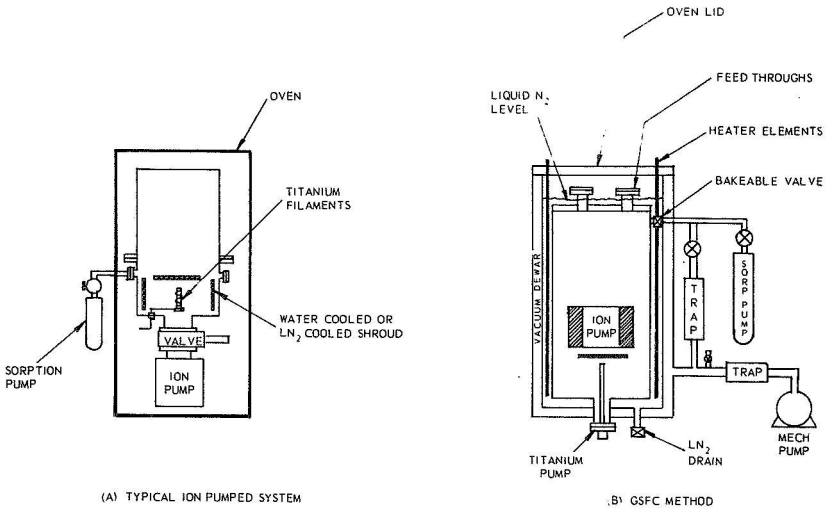

Fig. 1—Typical and GSFC ion pumping techniques.

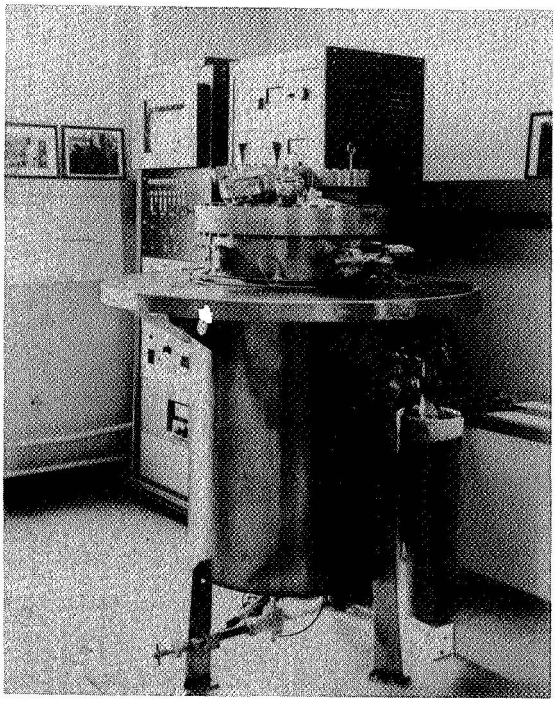

Fig. 2—GSFC experimental ultra high vacuum system.

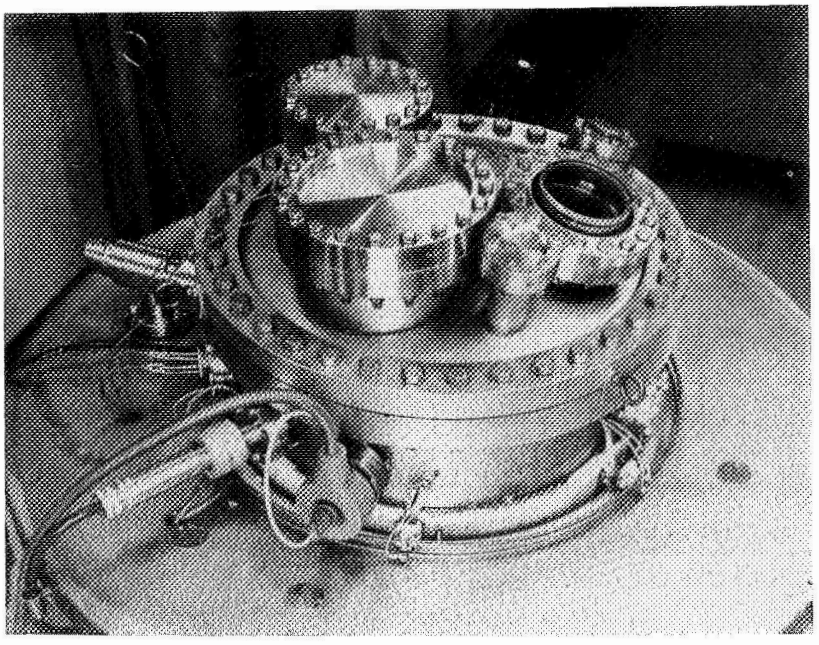

Fig. 3—Gage locations GSFC ultra high vacuum system.

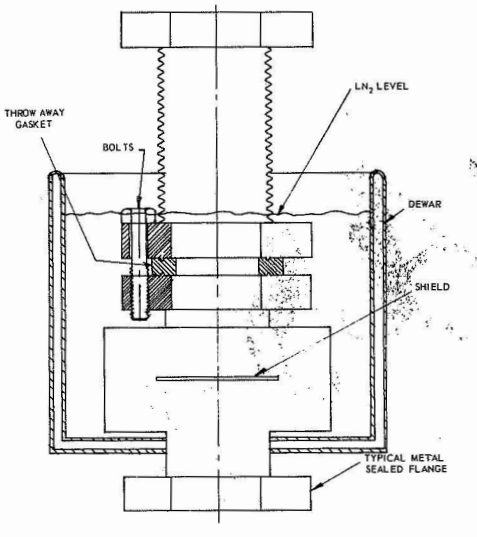

Fig. 4—Anti-migration trap.

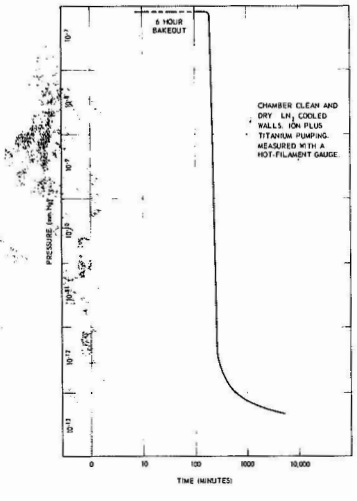

Fig. 5—Auger-spectrometer pump down cycle.